ULTRAFINE-GRAINED MATERIALS

Texture evolution of Mg during high-pressure torsion

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Abstract Polycrystalline magnesium of 99.8 wt.% purity
was subjected to high-pressure torsion (HPT) at room
temperature. A special technique was developed in order to
enable HPT of Mg up to very high shear strains of 115 and
hydrostatic pressures of 4 GPa. The texture development
during HPT was investigated by systematic X-ray texture
measurements. It can be described by a stationary oblique
B fibre characteristic of hexagonal metals subjected to
simple shear. From the measured shear strain and pressure
dependences of the *B* fibre and from microstructure
investigations by TEM, it is concluded that also mecha-
nisms of both dynamic *and* of static recrystallization
contribute to the texture evolution.

Introduction

Magnesium and its alloys have been extensively studied in
recent years, not only because of potential applications as
light-weight engineering materials, but also due to their
ability of hydrogen storage. Enhancing the strength with

simultaneously keeping sufficient ductility can be obtained
by methods of Severe Plastic Deformation (SPD) [1].
Among them, high-pressure torsion (HPT) allows to
achieve very high plastic strains due to enhanced hydro-
static pressure which prevents the deformed material from
cracking critical for deformation of pure Mg at room
temperature. Moreover, HPT allows precise controlling of
process parameters like shear strain, temperature and
hydrostatic pressure [2]. Considering the hitherto published
literature, the texture evolution of pure Mg in torsion has
been studied in detail, but only to shear strains of 0.9 [3].

The present work aims at systematically investigating
the texture of pure Mg subjected to HPT, i.e. up to very
high shear strains and to study the influence of hydrostatic
pressure. Microstructural investigations by Transmission
Electron Microscopy (TEM) were undertaken in order to
substantiate the findings resulting from texture analysis.

Experimental

Materials

Polycrystalline magnesium samples of 99.8 wt.% purity
were investigated after deformation by HPT. The starting
material for HPT was Mg extruded at 350 °C having an
average grain size of ~ 20 μm and a strong $\langle 0001 \rangle$ fibre
texture in extrusion direction. Disc shaped samples suitable
for HPT were cut by spark erosion from the extruded rod
normal to the extrusion direction. The diameters of the
torsion samples were 8 and 10 mm, their heights were 0.9
and 2 mm, respectively. In addition a 1-mm wide Cu ring
was attached to the torsion sample to prevent unstable
deformation caused by the excessive flow of the material
across the HPT plungers [2].

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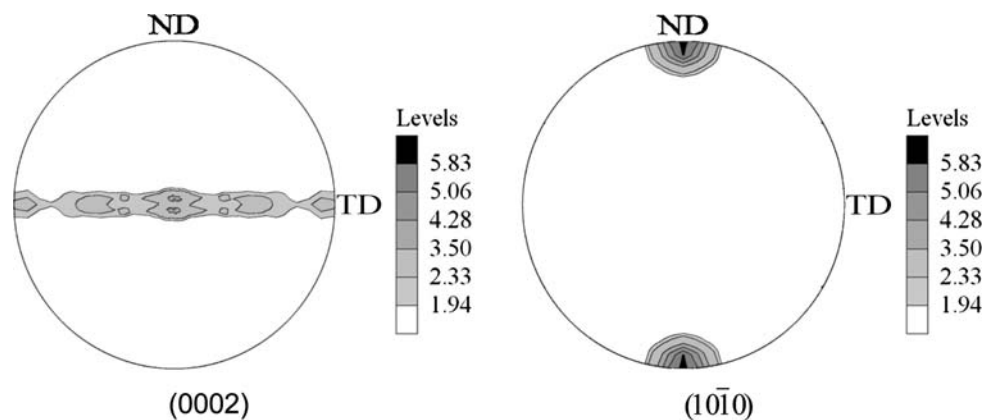1062 Dresden, Germany

High-pressure torsion

The HPT experiments were carried out at the Erich Schmid Institute for Material Science in Leoben (Austria). In situ flow curves of HPT were recorded during the deformation process by means of continuous torque—twist measurements. The
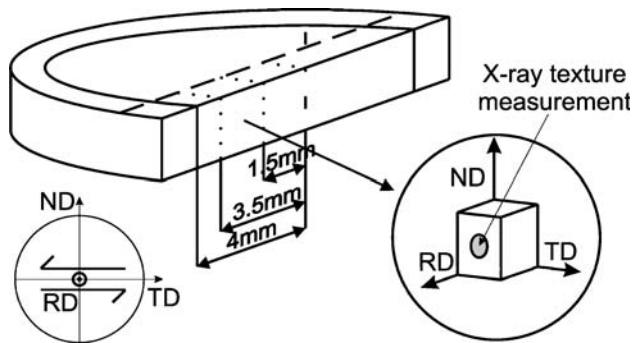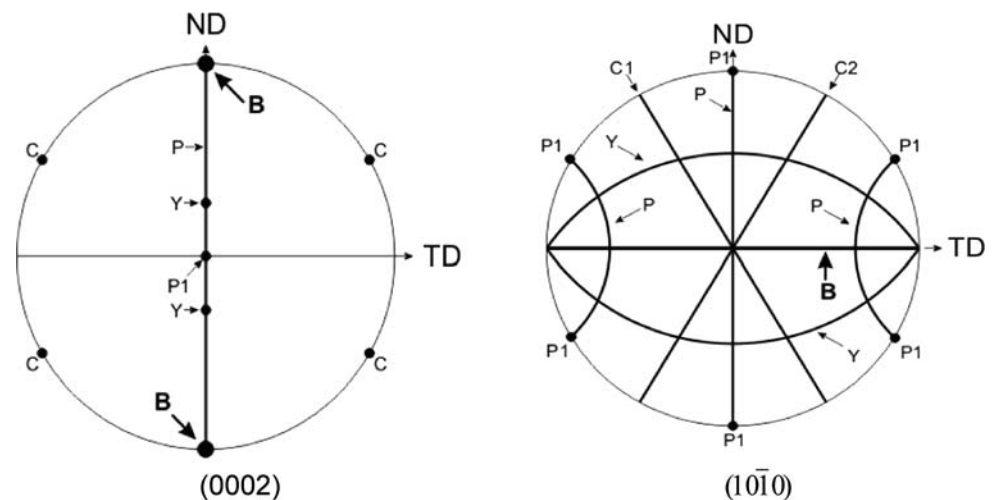

Fig. 1 Sample preparation for X-ray texture measurement of a HPT-deformed sample. ND, TD and RD refers to Normal Direction (torsion axis), Tangential Direction and Radial Direction, respectively. The shear direction is parallel to TD

Mg samples were deformed at hydrostatic pressures of 1, 2 and 4 GPa up to a maximum shear strain of about 115. The rotation of the plungers was at a rotation speed of 0.2 rot/min yielding a constant maximum shear strain rate of $5.3 \times 10^{-1}\ s^{-1}$. Sample thickness reduction (due to compression) during HPT experiments amounted to less than 12%, e.g. the initial sample thickness of 0.89 mm was reduced to 0.77 mm after 1 rotation at 2 GPa. All samples were deformed at ambient temperature.

Texture measurements

The textures were measured on sections normal to the radial direction of the torsion samples at distances of 1.5 and 3.5 mm from the torsion axis (Fig. 1).

For X-ray diffraction the Cu Kα line ($\lambda = 0.1542$ nm) and a beam collimator of 0.3 mm in diameter was used. Pole figures (0002), ($10\bar{1}0$), ($10\bar{1}1$) and ($10\bar{1}2$) were registered using a GADDS area detector being part of the Bruker AXS D-8 wide angle diffraction system. The Orientation Distribution Function (ODF) was calculated by means of the ADC method using monoclinic symmetrization within the

Fig. 2 Texture of Mg before HPT (reference coordinate system as shown in Fig. 1)

Fig. 3 Ideal orientations of Mg after deformation by simple shear as seen on (0002) and ($10\bar{1}0$) pole figures, with the B fibre emphasized ([3], see also there for nomenclature). For reference coordinate system see Fig. 1

Fig. 4 Texture evolution of HPT deformed pure Mg as a function of shear strain at three hydrostatic pressures represented as (0002) pole figures (reference coordinate system as shown in Fig. 1)
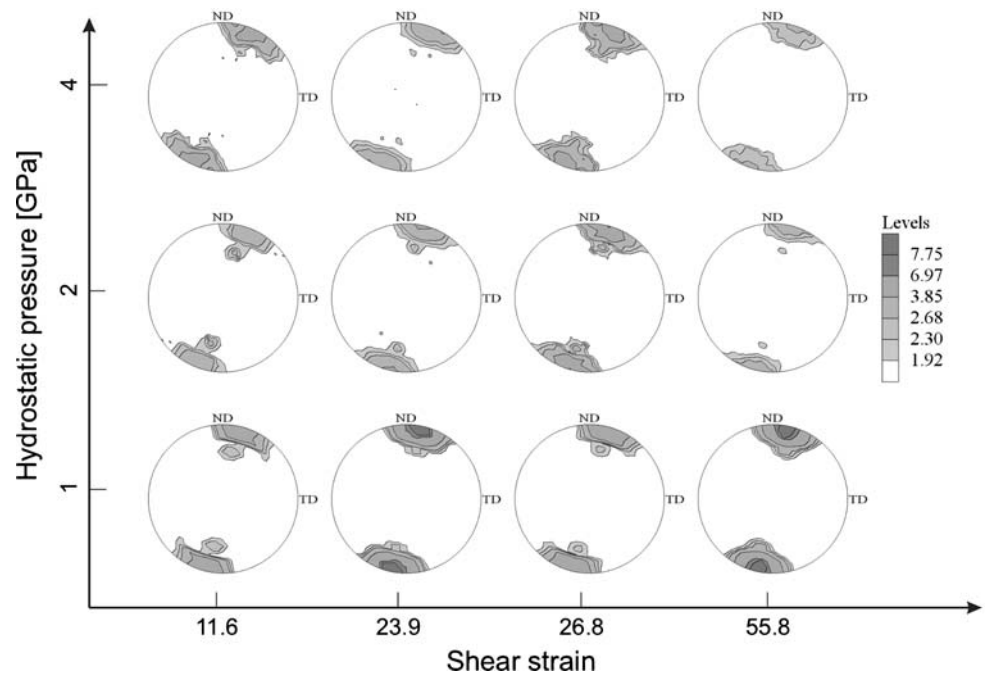
Fig. 5 Texture evolution of HPT deformed pure Mg as a function of shear strain at three hydrostatic pressures represented as $(10\bar{1}0)$ pole figures (reference coordinate system as shown in Fig. 1)
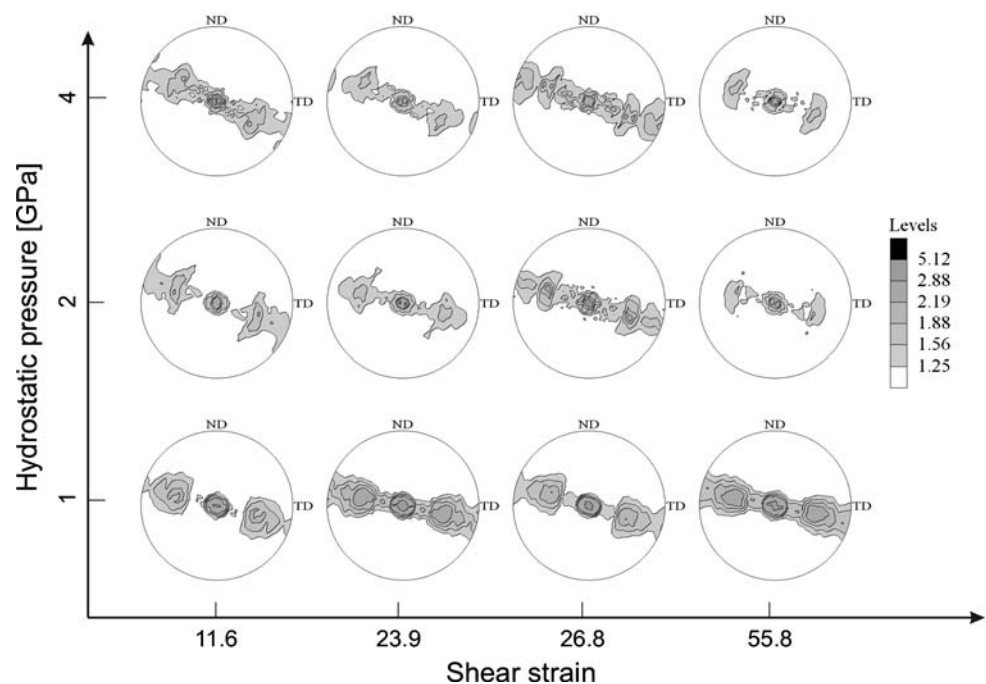
LABOTEX texture analysis software. The pole figures shown are recalculated from the ODFs. The fibre intensities in terms of volume fractions are calculated from the ODFs using a half width (FWHM) of 10°.
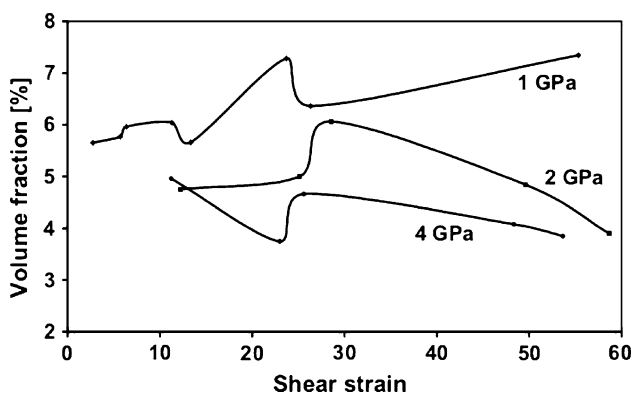
TEM investigations

For TEM specimen preparation, the HPT samples were mechanically ground to a thickness of about 0.15 mm. Then,

discs with a diameter of 2.3 mm were punched and double-jet electropolished to perforation using an electrolyte consisting of 1% perchloric acid and 99% ethanol. TEM investigations were carried out with a Philips CM200 electron microscope at an acceleration voltage of 200 kV. Since the microstructure in all TEM foils consisted of a mixture of areas with large, recrystallized grains and smaller as-deformed ones, it was tried to select representative sites in all samples and to image them in bright- and dark-field mode.
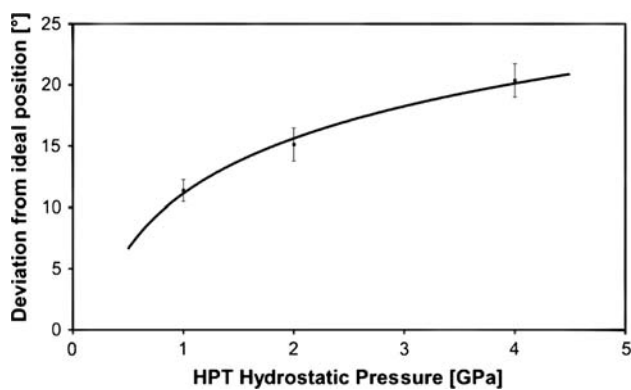
Fig. 6 Deviation of the B fibre from the ideal position as a function of hydrostatic pressure applied during HPT, as derived from the (0002) and (10$\bar{1}$0) pole figures presented in Figs. 4 and 5
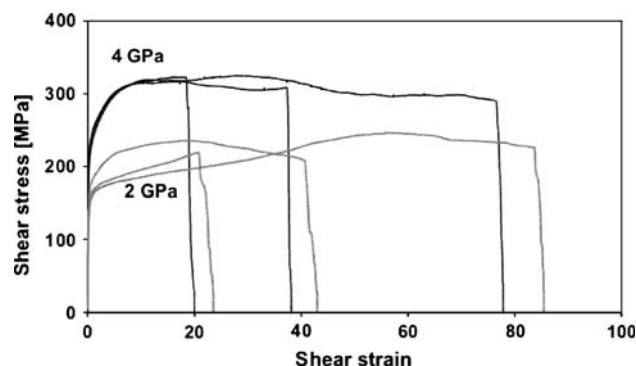
Results and discussion

During torsion of Mg the c axis initially aligned in the shear plane (Fig. 2) mainly rotate towards the torsion axis forming the so-called B fibre (Figs. 3–5). Details of the experimental texture evolution will be given elsewhere. Such a rotation has been described recently by Beausir et al. [3] who investigated the texture evolution during free-end torsion of pure Mg up to shear strains of 0.9. Simultaneous texture simulations with the Taylor visco-plastic crystal plasticity model using basal, prismatic and pyramidal slip with relative critical resolved shear stress values of 1, 8 and 6, respectively, confirmed this rotation.

However, the absolute value of the rotation was somewhat higher than that in experiment and for shear strains of

Fig. 7 Volume fractions of the B fibre as a function of shear strain arising from HPT at different hydrostatic pressures

Fig. 8 In situ monitored deformation curves of Mg during HPT at hydrostatic pressures of 2 GPa (lower grey lines) and 4 GPa (upper black lines)
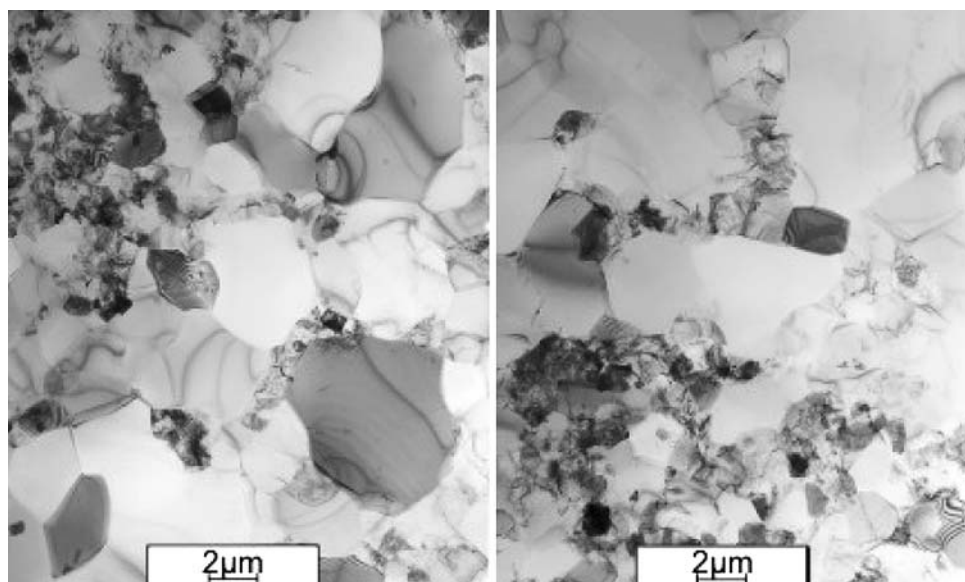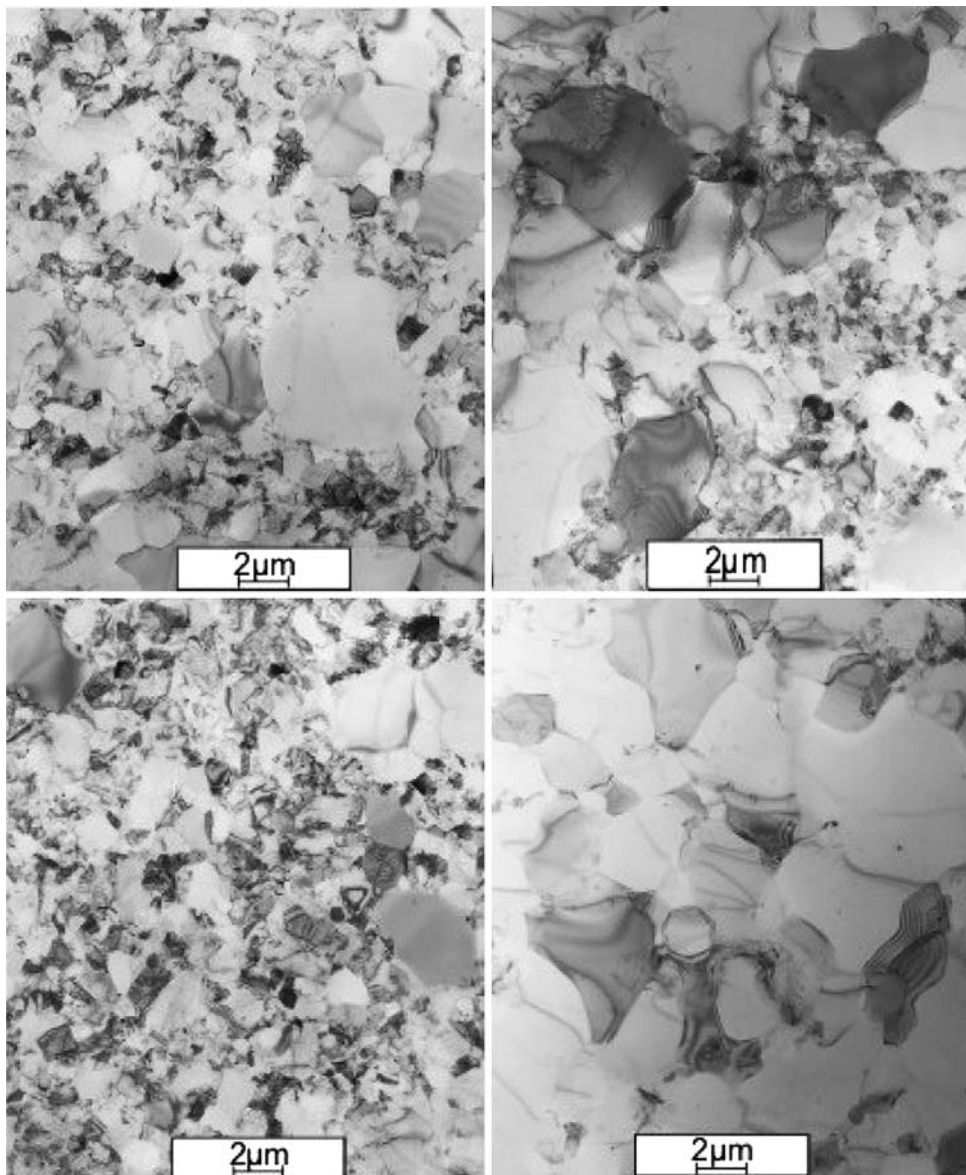

Fig. 9 TEM bright field images of HPT-deformed pure Mg up to a shear strain of about 25 at pressures of 2 GPa

about 8 even slightly exceeded 90°. In contrast, the textures in our work measured at shear strains larger than 10 exhibit a steady state behaviour with respect to rotation of the *c*-fibre axis as well as to texture intensity (Figs. 6, 7). The rotation of the fibre *c*-axis is only 80° or less (corresponding to a deviation from ideal position of 10° or more), depending on the hydrostatic pressure applied (Fig. 6). Following Skrotzki et al. [4], this indicates some process to occur which prevents the rotation of the *c* axes in reaching their ideal position. The limitation of rotation is suggested to arise from the onset of discontinuous dynamic recrystallization beyond a certain critical strain reached. Since the texture rotation found is the same for all strains

investigated, this critical strain is thought to be smaller than the smallest deformations of samples being subjected to texture investigation. With increasing hydrostatic pressure, however, the deviation of the *B* fibre from the ideal position becomes larger (see Figs. 4, 5 and especially Fig. 6); at the same time, the texture intensity decreases (Fig. 7). Following the argumentation above, these findings indicate a lowering of the critical shear strain for recrystallization with increasing hydrostatic pressure. This conclusion is confirmed if one inspects the characteristics of in situ monitored stress–strain curves shown in Fig. 8. Here it is seen that the onset of steady-state being represented by constant or slowly decreasing stress indeed occurs at lower

Fig. 10 TEM bright field images of HPT-deformed pure Mg up to a shear strain of about 115 at pressures of 2 GPa (left column) and 4 GPa (right column). Each state is documented by two images

strains if the hydrostatic pressure is increased. These in situ stress–strain curves also give evidence for the *dynamic* nature of recovery/recrystallization since absolute softening occurs in almost all curves, irrespective of the hydrostatic pressure applied (Fig. 8).

In order to demonstrate that recrystallization has taken place, the microstructure was investigated by TEM. All samples investigated at shear strains greater than 10 showed a recrystallized bimodal grain structure with equiaxed grains of about 1 and 5 μm (Figs. 9, 10) which confirms the interpretation of texture investigations. However, with increasing hydrostatic pressure, the volume fraction as well as the size of the recrystallized grains is markedly increased (Fig. 10). This observation does *not* combine with the assumption that recrystallization occurs purely *dynamically*, because in this case the grain size of recrystallized grains should decrease with increasing steady state flow stress which has been found in the in situ flow curves shown in Fig. 8. Moreover, previous investigations on the effect of pressure on recovery and/or recrystallization processes [5–7] showed that an increase of the hydrostatic pressure increasingly suppresses the mechanisms of diffusion in the material and also those of dynamic recovery and/or recrystallization, but enhances those of the static one due to increased driving force given by a high density of accumulated vacancies. Thus, in the present case recrystallization or at least some abnormal grain growth may have taken place during unloading, that means during and after release of the pressure. Usually, grain growth does not change the texture drastically.

At this point of discussion we are left with the conclusion that both static *and* dynamic recrystallization processes might contribute to the texture effects found in HPT deformed Mg. For a quantitative determination of these contributions further investigations appear to be necessary.

Summary

The strain evolution of texture and microstructure in pure Mg deformed by HPT at different hydrostatic pressures can be described as follows:

- Pure Mg subjected to HPT develops a steady state texture, the strength of which depends on the hydrostatic pressure.
- The texture is mainly characterized by the so-called *B* fibre typical of simple shear deformation of hexagonal metals. However, the fibre axis deviates from its ideal position against the shear sense. The angular deviation increases and the texture strength decreases with the hydrostatic pressure applied.
- TEM investigations show a recrystallized microstructure consisting of a bimodal grain size with the volume fraction and size of larger grains increasing with hydrostatic pressure.
- In addition to the typical shear components, textures and microstructures can be described best in terms of discontinuous *dynamic* as well as of discontinuous *static* recrystallization mechanisms.

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